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oddre ltr, 13 apr 1971

Third Progress Report

by the

Committee on Tubing

of the

Materials Advisory Board



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National Academy of Sciences— National Research Council Washington, D. C.

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"... to conduct studies, surveys, make critical analyses, and prepare and furnish to the Director of Defense Research and Engineering advisory and technical reports, with respect to the entire field of materials research, including the planning phases thereof."

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THIRD PROGRESS REPORT

COMMITTEE ON TUBING

Prepared by the

Materials Advisory Board

Division of Engineering - National Research Council

The National Academy of Sciences
and
The National Academy of Engineering
to the
Office of Defense Research and Engineering
Department of Defense

as a service of

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National Academy of Sciences—National Research Council
Washington, D. C.

November 1966

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This report is one of a series in a study undertaken by the Materials Advisory Board for the National Academy of Sciences in partial execution of work under Defense Supply Service Contract Number DA-49-083 OSA 3131, between the Department of Defense and the National Academy of Sciences.

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ABSTRACT

This report, the third in a series, summarizes the presentations made at a meeting of the Committee on October 6, 1966. A number of discrete topics were covered, principally tubing requirements for radioisotope devices, differences between various Ta-W-Hf alloys, and specifications for refractory metal tubing.

The status of contracted research by the Air Force, AEC, NASA, and Army is reported. The alloys involved include TD Nichrome and various compositions based on V, Cb, Ta, Mo, and W.

REPORT OF THE MEETING WITH REPRESENTATIVE OF THE OFFICE OF SALINE WATER

Mr. Goldberg reported on a discussion held, along with an MAB representative, with Mr. Paul Tomalin. The purpose of this discussion was to inquire whether OSW wished to make any use of the Tubing Committee. Mr. Tomalin described his method of operation, which involves making extensive use of trade groups and ASTM for advice and the Oak Ridge National Laboratory for engineering support. The eventual demand for the very large quantities of tubing tends to make the tubing industry very cooperative. Therefore, Mr. Tomalin felt no need to turn to this group.

Mr. Goldberg called attention to an article on Page 29 of the September 5, 1966, issue of "Steel" magazine, which gives an excellent description of the desalination program.

REPORT ON TUBING REQUIREMENTS FOR RADIOISOTOPE DEVICES

Dr. Topper of the Atomic Energy Commission commented first that his remarks were unclassified and that new rules of classification permitted open discussion regarding materials for use below 1200F.

In a general review of devices, it was stated that plutonium 238 is generally regarded as the most desirable isotope. The heat generated is converted to lead telluride thermocouples into electricity. Haynes 25 is the material commonly used for capsules when the temperature is below 1200F. The desirable attributes for capsules are:

resistance to sea water corrosion, air oxidation resistance, containment of helium pressures, impact strength.

Haynes 25 is limited in temperature capability, and refractory metals must be turned to at the higher temperatures. These metals must be clad, for which purpose noble metals and alloys are being looked at most seriously. Tantalum is the usual first choice of the refractory metals. Helium venting is of much interest to the Commission but this is a design, not a material, problem. Other isotopes such as strontium are easier in every way to design for; there is, for example, no gas containment problem. On the other hand, there is a shielding penalty with these other isotopes. Plutonium 238 is scarce and expensive, while strontium is still unpopular, largely for political-psychological reasons. For systems exceeding a power of about 500 watts, plumbing would be needed through which NaK would circulate, as with SNAP-8. Fabrication of a capsule is carried as far as possible before loading the isotope, but final closure must be done with the isc ope inside. Practical considerations require that construction must be consistent with Mound Laboratory practices for capsule sealing. An increase in production of radioisotope devices is anticipated which will not require a large tonnage of material, but an amount which might be significant in terms of refractory metal tubing production. The

requirement for long operating lifetime will require that material will have to be ordered to special specifications, and stringent quality control will have to be applied. A decision regarding the optimum alloy has not been made.

T-111 FAMILY OF ALLOYS

In response to questions indicating confusion in the minds of users of these alloys, Mr. Begley summarized the characteristics and compositional differences. T-111 originated as a solid solution alloy with a low interstitial content (less than 40 ppm each of 0, N, and C). The basic composition is Ta-8W-2Hf. T-222 has the composition of 9.6W-2.4Hf and 0.01C. The newest alloy "T-333" has been given the name Astar 811C (Ta-8W-lRe-0.7Hf-.025C). The difference in properties reported is attributal to two principal factors: test atmosphere and metallurgical structure. Both of these factors are more important in creep than in tensile testing. Data were presented to illustrate the fact that metallurgical structure, as modified by prior thermal-mechanical history, has a very pronounced effect on creep properties. T-111 is generally more forgiving than other alloys, particularly regarding poor welding practice. Because of higher creep strength, Astar 811C would be the choice if weight were critical. The small amount of rhenium in Astar improves the creep strength greatly but has no other effect.

JOINING OF BIMETAL TUBING

Mr. Begley made a brief report on a program nearing completion. In this work, stainless tubing with an inner core of columbium was joined by several methods. The purpose was to answer the question "Can we make reliable joints using state-of-the-art techniques suitable, in the case of butt welds, for field operation"? As a result of the program, it was concluded that all three types of joints attempted (butt, tee, and tube-to-header) could be made successfully. The real problem described in this study was with the quality of the material itself (wall thinning, laps, seams and debonded areas). Nondestructive evaluation of final closure is also a problem. Ten of each type of weld were made and are in the final stages of evaluation.

The work was done under contract NAS-3-76-21 at the Westinghouse Astronuclear Laboratory.

AIR FORCE PROGRAM REPORT

Mr. Glenn provided the following information on a recent contract with duPont, "Dispersion Strengthened Nickel - Chromium - Thoria Alloy: Tubing and Bar."

The primary objectives are to define a chemical alloy composition and to develop process sequences which are adaptable to production equipment and which will yield strong tube and bar in a limited size range. A secondary objective is to understand why a given process route yields higher strengths at the goal size so that process routes can be extended to additional sizes.

The approach is to determine the optimum composition and investigate processes for the consolidation, sintering and conversion of 6- and 8-inch diameter billets to bar and tube blanks to be reprocessed to thin wall, small diameter tubing. Bar process routes to be investigated include bar rolling, double extrusion to bar, and extrusion to intermediate sizes followed by GFM rotary swaging. For the tubing effort, extrusion processes will be developed to produce 1-1/2 inch 0. D. by 3/16 inch wall tube blanks. The tube blanks are to be converted to a goal size of 1/8 inch 0. D. x 0.010-0.012 inch wall thickness by means of warm and cold drawing and cold tube reduction; combinations of these tube routes, such as warm drawing followed by cold tube reduction, will be investigated as well as recrystallization heat treatments and their proper placing in the process sequence.

The tubing and bar work will include evaluation of the material fabricated in the fine-grained and in the recrystallized or partially recrystallized condition. There probably is an optimum amount of secondary work required prior to final heat treatment. Determining this optimum amount of secondary work, the most favorable structure to apply to it, the proper placement of in-process heat treatments, and the correct heat treatment conditions will be among the primary tasks of the program.

Phase I has involved the determination of optimum alloy chemical composition and the investigation of processes for converting compacted and sintered billets to various bar sizes and to tube blanks for reprocessing to tubing. A nominal composition of Ni-20Cr-2ThO₂ (TD NiC) has been chosen for further development. A process has been developed to produce good quality tube blanks of 1-1/2 inch O. D. x 3/16 inch wall. The compacted and sintered billets are first extruded to fully dense, four-inch diameter billets which are then re-extruded to tube blanks. The double extrusion process allows the bare extrusion to tube blanks without the problem of internal contamination which would be encountered in direct extrusion of non-dense billets to tube blanks. Tube blanks have been supplied to the tubing subcontractors, Wolverine Tube and Superior Tube. Cold tube reduction to 7/8 inch O. D. x 0.083 inch wall thickness has been accomplished. TD NiC is smenable to cold tube reduction provided that the

outer surface is exceptionally smooth with no circumferential scratches or notches. Several tube blanks have been warm drawn at about 1200F through a light pass (less than 10% reduction in area). The surface after drawing is good. The goal for tubing manufacture is to maintain the high-temperature strength levels obtained on recrystallized extruded tube blanks.

The as-extruded structure of TD NiC shows a very small grain size of 1-5 microns which will recrystallize to a grain size of about 0.1 mm or larger, with a post-extrusion heat treatment of one hour at 2400F. After recrystallization, the 2000F strength increases by a factor of about 2.8. Prior to recrystallization, the strength at 2000F is about 5000 psi with ductility values of 50% R. A. or more, and per cen elongation of over 100%. Because the fine-grained structure is very ductile, there is an incentive to maintain this fine-grain structure as far as possible during processing, as in the case where an extrusion is not a final product but merely an intermediate shape.

TD NiC bar stock for secondary fabrication has been produced by extruding to six-inch diameter, billets of 3/4 inch to 1-1/2 inch diameters, at extrusion temperatures of 1600F to 2200F, employing glass lubricants, (CEFILAC). Good 2000F properties are obtained in extruded bar after recrystallization. Secondary GFM forging has increased the elevated temperature properties over the as-extruded and recrystallized material.

Phase I is still in progress. Future work will include scale-up to 8-inch diameter billets of 20-inch lengths. Secondary fabrication techniques such as bar rolling, GFM rotary forging, and double extrusion to bar will be evaluated. As with TD NiC tube, metallurgical studies to determine the role of structure in the strengthening of this alloy will be continued.

Pre-alloyed powders have been hydrostatically compacted and extruded to dense stock for reprocessing. Two 8-inch diameter billets have been extruded to dense re-extrusion stock. Thirteen lengths of tube blanks extruded earlier are now being conditioned preparatory to the Phase I tubing manufacture. Five bars have been GFM rotary forged. Two additional 6-inch diameter billets were extruded to 3/4-inch diameter and 1-inch diameter bar. Two additional 6-inch diameter billets were extruded to 3/4-inch diameter and 1-inch diameter bar. These bars have good combinations of high temperature strength and ductility.

Mr. Glenn introduced Mr. F. A. Glaski of San Fernando Laboratories who described work for the Air Force. Under this contract, internal cladding of stainless steel and other types of tubing ranging from 0.250" to 4" in diameter and 10' to 15' in length have been successfully fabricated where the internal clad material has been tungsten, tantalum, columbium or Cb-1% Zr. The latter two materials have been investigated under Air Force

Contract AF 33(615)-2226 and have been deposited on the inner surface of 316 ss tubing lengths up to 12' long. These lengths have been both constant diameter (0.8" i.d.) and abruptly changing diameter (from 0.8" i.d. to 2" i.d. and back to 0.8" i.d.). Two 7' x 4½' 316 ss loops have been scessfully coated on their interior surfaces with Cb-1%Zr. One of these loops, as well as a third loop that has yet to be coated, will be delivered to Wright-Patterson AFB for testing in a boiling potassium environment. Constant diameter boiler tubes with Cb-1% Zr swirl tape inserts will also be internally clad with CVD Cb-1% Zr and these coated tubes will be delivered to Wright-Patterson for evaluation testing.

The heating method reported earlier was incremental and resulted in hydrogen embrittlement, which caused a poor bond. Plating the entire assembly at a uniform temperature, which is now done, eliminates this problem. The problem of depositing a uniform thickness from end to end is solved by use of low pressure, an argon diluent, adjustment of gas flow, etc. The time required to deposit a 6-7 mil coating in a 12-foot tube is of the order of two hours. No trouble with delamination appears even when a 1/2" tube is bent around a 6" radius. In the case of tungsten deposits, the structure after recrystallization consisted of fine equiaxed grains. Photomicrographs of W-20 Re deposits were shown in which a chevron grain structure appeared.

Other work of interest not sponsored by the Air Force, includes production of free-standing thermocouple sheath sized tubing (0.040" - 0.125") diameter x 6' - 7' long made of tungsten, molybdenum, tantalum, and tungsten/20% rhenium alloy. The tungsten/20% rhenium alloy possesses a wrought-recrystallized, equiaxial, fine grain structure and possesses a measure of room temperature ductility. A similar structure, with an equiaxial grain size of 80,000 grains/mm² or finer, demonstrated to remain stable after thermal treatment at 2300C for one hour, has also been reproducibly manufactured in pure tungsten tubing. Internal cladding of 9Cr-1Mo steel tubing, and of stainless steel header assemblies with tantalum was also reported. The assemblies have typically consisted of a number of

3/8" diameter x 3^{1} x 6^{1} long tubes welded into a 3/8" diameter header at either end.

Mr. Glenn concluded by stating that two recently completed programs have generated tubing now in inventory which might be available on request. From the duPont D-43 contract 1/8" tubing (15 mil wall) and 1/4" tubing (30 mil wall) is on hand. The Allegheny-Superior contract has produced Ta-10W and Ta-222 in 1/4", 3/8", and 1/2" diameters.

ATOMIC ENERGY COMMISSION PROGRAM REPORT

Atomic Energy Commission requirements for refractory metal tubing appear to be disappearing rapidly. The potential for civilian power reactors seems much greater than that for the space programs. However, operations are continuing on a much reduced scale to obtain property data and to retain an in-house ability to produce material. In the thermionic area the major interest is in deposited tubing but here it is believed that a commercial product is available.

The contract with 3M has been terminated. Most of the objectives were met with the possible exception of as-sintered wall thickness. Tube lengths of 15 inches or more in small diameter were produced to requirements. The requirement of 95 percent theoretical density proved easy to meet. Some of the tubes were drawn successfully at Argonne. Evaluations of properties such as weldability will be done on a purchase basis.

At GE-NPO, extrusions have been made in which the Mo filler incorporated a small rod of Hf. The Hf dissolves readily, permitting easy dissolution of the Mo. GE production can satisfactorily meet requirements of tubing for test purposes. This year they will compare arc cast with sintered material and will also examine changes aimed at making the process more economical. The properties of binary and ternary alloys will be evaluated.

Vanadium alloy tube development being carried on at Argonne is intended as a backup in the fast breeder program for the stainless steel which is now the top candidate for a cladding material in contact with liquid sodium. Mr. Mayfield reported that no particular difficulties have been experienced in processing or fabricating the alloys, but emphasized that the quality of the finished tubing is highly dependent upon the quality of the extruded or tube reduced blanks.

Mr. Harms, referring to the Oak Ridge work for AEC, mentioned two main approaches to cost cutting: 1. Extrusion over a floating mandrel (over a layer of molybdenum) rather than the filled billet technique, and 2. Vapor deposition of unalloyed tungsten and W-Re alloys.

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Mr. Milko stated that his interest was in a Plumbrook Rankine system. Tubing for this purpose would be in the range of 3-7 inches diameter with wall thicknesses from 0.06"-0.25". The design has not advanced to the point of indicating preferred sizes.

Mr. Ault distributed a rough draft of a report entitled "Extrusion of 1/2- and 3/8-Inch Diameter, Thin Wall Tungsten Tubing Using
the Floating Mandrel Technique," by C. P. Blankenship and C. A. Gyorgak.
As a result of the program, usable lengths to 5 feet have been extruded.
The properties obtained were comparable to those of sheet and bar products. From another study where tubing had been extruded from W containing dispersed oxide particles, he reported that fibering of the oxide
particles occurred, producing L/D ratios of about 200. Unfortunately
these were not oriented to be usable in imparting additional hoop strength
to the tubing, but did increase the longitudinal strength.

ARMY PROGRAM REPORT

The U. S. Army Materials Research Agency is conducting a program to develop technology for production of small arms barrels with refractory metal liners. Experimental liners of a molybdenum alloy fashioned by the "3M Process" (a powder metallurgical technique) have been procured from the 3M Company, St. Paul, Minnesota. The tubular liners possessed a density of 87.7% as sintered. Dimensional check of the liners showed generally good compliance with target tolerances. Occasional voids were detected in the liners by radiographic inspection.

Several experimental liners have been swaged into short lengths of AISI 4140 steel barrel, an operation which imparts lands and grooves to the bore surface. The first composite barrels assembled in this fashion presented a very good internal appearance on visual inspection with a boroscope and met go/no-go measurements for compliance with dimensional requirements. However, after test firing, cracks were found to have developed in the liner material. Sectioning revealed that a loose mechanical bond was maintained which did not properly support the liner.

Experiments are continuing toward development of improved mechanical or metallurgical bonds between the liner and steel barrel.

In a project aimed at the same weapons development objective, Springfield Armory has procured some 100 feet of coextruded barrel stock from Nuclear Metals, Inc. Composite billets 5.5 D x 10.5 inches in size are extruded at a 14.6:1 reduction ratio at 1920F to yield approximately 100 inches of 1.4 D bar stock after cropping. The composite billet consists primarily of an exterior cylinder of AISI 4150 steel within which a sleeve of tantalum alloy 0.375 inches in thickness is placed. A central core rod of steel is inserted in the sleeve. The cylinder assembly is encased within a steel jacket which is evacuated and sealed prior to extrusion. The core material is removed from the extruded rod by selective etching.

The tantalum alloy liner is reduced to a thickness approaching 0.025 inches by extrusion. Microexamination of as-extruded composite reveals a metallurgical bond between liner and barrel steel.

Several sections of composite barrel have been cold swaged over a shaped mandrel to impart rifling with no evidence of operational difficulty. The pieces are under study to determine effects of swaging.

REPORT ON REVIEW OF SPECIFICATIONS

Mr. Krivetsky reported on the returns he had received after writing a number of organizations to obtain their tubing specifications. There is great diversity of items covered by these various specifications. The Magnaflux Corporation has made a summary of non-destructive test methods for tubing, which was made available to him. The subject of test procedures is not covered in any of the specifications. Mr. Krivetsky stated that he will soon be able to summarize the available information, permitting discussion on what action the committee may take.

AGENDA

TUBING COMMITTEE

Argonne National Laboratory October 6, 1966

9:00	a.m.	Welcome - Argonne National Laboratory	
9:10	a.m.	Report on Desalinization Meeting	D. Goldberg & J. Lane
9:20	a.m.	Report on Tubing Requirements for Radioisotope Devices	Leonard Topper, AEC
10:15	a.m.	Coffee Break	
10:30	a.m.	T-111 Family of Alloys and Joining of Bimetal Tubing	R. Begley, Westinghouse
12:00	noon	Lunch	
1:00	p.m.	Tour of Argonne National Laboratory Facilities	
		GOVERNMENT PROGRAM REPORTS	
2:00	p.m.	Air Force	George Glenn & F. A. Glaski
2:30	p.m.	NASA	John Milko & G. M. Ault
2:50	p.m.	AEC - W-Re-Mo and V Programs	A. Van Echo, W. Harms, & R. Mayfield
3:30	p.m.	Army	S. V. Arnold
3 - 45	n m	Report on Review of Specifications	A. Krivetsky

4:00 p.m. Adjourn

ATTENDEES

TUBING COMMITTEE

Cctober 6, 1966

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R. T. Begley, Westinghouse Astronuclear
Fred A. Glaski, San Fernando Laboratories
L. J. Goetz, General Electric
W. L. Hunsberger, Superior Tube
Michael V. Nevitt, Argonne National Laboratory

Leonard Topper, Atomic Energy Commission

Excerpt of Two P:ograms from GEMP-400A
"Fifth Annual Report-High Temperature Materials Programs"
covering the work period January 31, 1965 to January 31, 1966

General Electric Company Abstract

High-Temperature Reactor Materials Research

The purpose of this program is to measure and evaluate hightemperature physical and mechanical properties of cormercially available and newly developed refractory materials being considered for use in fueled and non-fueled high-temperature (1000 to 3000C) reactor applications.

During the first six months of CY-65, this task included both materials evaluations and materials research and development. During the last six months, the work was separated into two programs (1) "High-Temperature Reactor Materials Evaluations," Job 57003 and (2) "Refractory-Metal Alloy Research and Development," Job 57015 (Section 4 of this report). This section of the report will cover only the work performed on materials evaluations (principally mechanical property evaluations).

Refractory-Metal Alloy Research and Development

The over-all objective of this program is to advance the technology of refractory metals to permit the early utilization of these materials in high-temperature reactors. Specifically, the program encompasses development of new refractory-metal alloys to improve the temperature and life capabilities of nuclear power plants.

Creep-Rupture Properties of Tungsten and Tungsten-Base Alloys
H. E. McCoy

Oak Ridge National Laboratory
Contract No. W-7405-eng-26, August 1966

Abstract

The creep-rupture properties of two types of unalloyed tungsten and two tungsten-base alloys were determined at 1650 and 2200C for times of at least 500 hours. The materials studied were wrought powdermetallurgy tungsten, several lots of thermochemically deposited (TCD) tungsten, W-2% ThO2, and Sylvania A (W-0.5% Hf-0.02% C). At 1650C the order of strengths from the greatest to least was W-2% ThO2, Sylvania A, and TCD and powder-metallurgy tungsten. At 2200C the order of strengths from greatest to least was W-27 ThO, TCD and powder metallurgy tungsten, and Sylvania A. The ductility of the TCD tungsten was low at 1650C, but was good at 2200C. The W-2% ThO, showed moderate ductility at both temperatures. The ductility of the Sylvania A improved from moderate at 1650C to good at 2200C. The powder metallurgy tungsten had good ductility at both test temperatures. All of the materials studied exhibited better high-temperature grain-size stability than the powder-metallurgy material. Voids formed at the TCD tungsten at 2200C and grew to quite large sizes under an imposed stress. These voids were thought to be associated with impurities.

Highlights of GE-NMPO Research

January 1965 to July 1966

General Electric Company

Abstract

The objective of the high-temperature materials research and development program at GE-NMPO is to extend high-temperature reactor technology by developing new, improved materials and components as well as a better understanding of the physicochemical properties of these materials. More specifically, the areas being studied are (1) radiation effects in BeO heat-resistant metals and refractory metals, (2) refractory metal alloy development and the properties of refractory metals, (3) fission product transport processes in refractory metal fuel systems, (4) high-temperature thermocouple systems, (5) oxidation- and steam-corrosion resistant alloys and fuel elements, and (6) clad UO₂ in potential melt-down environments.

Strengthening Effects in Ta-W-Hf Alloys
Technical Paper Presented at AIME Symposium on
Physical Metallurgy of Refractory Metals
French Lick, Indiana, October 3-5, 1965
WANL-SP-013, January 15, 1966
Westinghouse Astronuclear Laboratory

Abstract

The work described in this paper was done in the course of a Tabase alloy development program sponsored by the Bureau of Naval Weapons. In an earlier survey of Ta-base alloys it was demonstrated that Ta-W-Hf solid solution alloys exhibit attractive high temperature mechanical properties in addition to excellent fabricability and weldability. Typical of alloys in this system is the Ta-8W-2Hf alloy designated T-111. The more recent work was aimed at optimizing the composition of T-111 with respect to strength and fabricability. This involved evaluating 1) the effect of interstitial additions of C, O, and N to T-111, and 2) the effect of increasing the substitutional solute content to 14 w/o (W + Hf) with W to Hf ratios ranging from 1.3 to 6. On the basis of the observed strengthening effects and the concurrent evaluations of weldability and fabricating characteristics, a final alloy composition was selected which reflected a modest increase in W + Hf content plus an intentional, but small, addition of C. This optimized alloy is designated T-222 and has the nominal composition Ta-10W-2.5Hf-0.01C.

In an effort to clarify the mechanism whereby interstitial solutes contribute to the high temperature strength of T-222 alloy, the identity and morphology of the second phases present in the alloy were studied for various mechanical-thermal histories. In the present paper, the results of tensile tests on the various alloys are summarized to demonstrate the strengthening effects observed, and then the results of the phase identification study are presented. Following this, an interpretation of the observed interstitial effects is presented.

Extrusion of 1/2- and 3/8-Inch Diameter, Thin Wall

Tungsten Tubing Using The Floating Mandrel Technique

C. P. Blankenship & C. A. Gyorgak

Lewis Research Center, NASA

Abstract

Extrusion of small-diameter, thin-wall tungsten tubing was investigated using the floating mandrel technique. Variables studied included extrusion temperature, initial density of the tungsten, and composition of the canning material. Mandrel diameters of 0.4 and 0.3 inch were employed in extruding the 1/2- and 3/8-inch diameter tubing, respectively. Nominal wall thicknesses were 0.020 inch. Extrusion temperatures ranged from 3200F to 4000F. The extruded tubing was examined for dimensional variations, surface finish, density, hardness, and microstructure. Machanical properties were determined at 3500F under tensile and biaxial stress conditions.

This paper is in rough draft form.

Correction

An error appeared in Second Progress Report (MAB-219-M(2)). The third paragraph on page 1 should be corrected to read " . . . pH 6.8 and temperatures up to $250^{\circ}F$."

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